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TECHNICAL REPORT AFOSD-TR-78042

IMPROVED ADHESIVE BOND PERMANENCE WITH
REACTIVE ORGANOSILANE COUPLING AGENTS

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US ARMY ARMAMENT RESEARCH AND DEVELOPMENT COMMAND
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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) → Five reactive organosilane coupling agents, (Dow Corning Z6020, Z6032, Z6040, Z6062 and Union Carbide Corp. A-1100) were evaluated as adhesion promoters to improve the adhesive bond strength of 2024T3, 6061T4 aluminum alloys and 302 stainless steel and to protect the bonds against the deleterious effects of water. The organosilane coupling agents, mercaptopropyltrimethoxysilane (D.C. Z6062), and glycidoxypropyltrimethoxysilane (D.C. Z6040) generally showed better bond strength → over		

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initially and after adverse exposure than the control samples when bonded with room temperature-curing epoxy resin. The glycidoxypopyltrimethoxysilane (Z6040) showed better initial bond strength than the control samples and good retention of bond integrity after adverse exposure when used with aluminum alloys and an intermediate cure temperature [121°C (250°F)] epoxy adhesive. The remaining organosilanes did not show appreciable improvement over the control samples.

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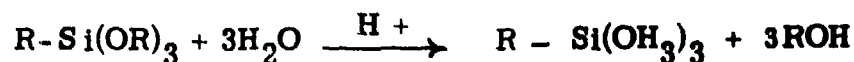
INTRODUCTION

Silane coupling agents (ref 1 and 2), because of their ability to bond organic polymers to inorganic materials, can be used to improve the adhesive bonding of metals. The hydrolyzable groups on silicon are generally believed to be the means by which the silane coupling agents interact with the metallic oxides. The silanes form chemical bonds at the interface that maintain high strength under high humidity and other adverse environmental conditions. The organic moiety of the silane reacts with organic functional groups of the adhesive. Thus, the organosilane functions as a true coupling agent, bonding the inorganic metal oxide to the organic adhesive. A true coupling agent bridges the interface.

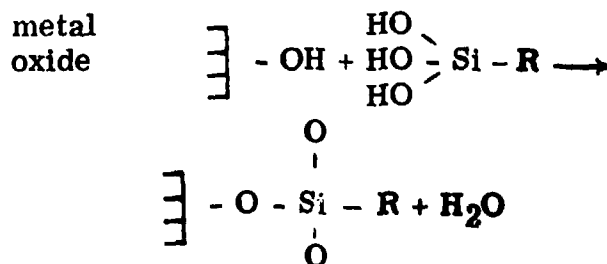
The hypothesized mechanism is:

Silane-to-Inorganic Surface Interface

1. Hydrolysis



2. Equilibrium Bond Formation

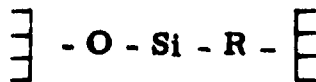


Silane-to-Organic Polymer Interface

The formation of covalent bonds between the silane's organo-functional moiety and reactive species of the polymer matrix is:



metal
oxide



organic
resin

Among the more reactive organic groups available on silane coupling agents are vinyl, alkyl amino, alkyl mercapto, and epoxy radicals (table 1).

DISCUSSION OF RESULTS

In this investigation, five different reactive organosilanes (table 1) were evaluated as adhesion promoters to improve bond strength and protect the bonds against the deleterious effects of water. Initial experiments were run to determine the conditions to be used to evaluate these coupling agents. A summary of the results is given in table 2. Individual results are given in the Appendix.

1. Using a methanol wash followed by compressed air-drying of the FPL-etched specimens resulted in an improvement of approximately 10% in lap-shear tensile strength.
2. Untreated acetone-degreased specimens coated with organosilane solutions had lap-shear tensile strengths much lower than the FPL-etched control specimens.
3. Varying the concentrations (0.1, 0.5, 1.0%) of the organosilane solutions did not appreciably alter the bond strengths (table 2).
4. The method of applying the silane solution to the FPL-etched specimens (brush-on or dip) did not appreciably affect the strength of the bonds (table 2).

The amino and vinyl functional organosilanes did not show any improvement in bond strength over the control samples when 2024T3 aluminum specimens were bonded with Epon 828/Versamid 140. In several experiments they had lower bond strengths. The vinyl functional silane did not react with the epoxy group. There is some indication that aluminum specimens containing high concentrations of copper render amino functional silanes ineffective

(table 2).

The mercapto and epoxy organosilanes had bond strengths 43% and 29% greater, respectively, than the control samples (Appendix). After a 34-day (816-hr), 60°C (140°F) water soak, the mercapto silane bond strength was 41% and the epoxy silane 43% better than the control water soak.

Experiments in which the organosilane was added directly to the adhesive resin indicated essentially no improvement over coating the 2024T3 aluminum adherends with silane solution initially or after a 34-day (816-hr) hot water soak (table 2). Experiments in which the mercapto silane specimens were bonded by coating the adherend and adding the silane to the resin resulted in stronger bonds than the uncoated control, however, not as strong as the bond formed by coating the adherend only. In a similar experiment using epoxy-functional silane, the bond strength was greater than the control or coating the adherend solely.

EXPERIMENTAL PROCEDURE

Preparation of Adherends

2024T3 and 6061T4 aluminum alloys were etched using the FPL etch before bonding. The panels were degreased with acetone and treated by immersing the bonding surface in a solution containing 1 part by weight (pbw) sodium dichromate, 10 pbw concentrated sulfuric acid, and 30 pbw deionized water. The solution temperature was 60°C (140°F) and the immersion time 7 to 10 minutes. The treated panels were rinsed for 1 to 2 minutes in running tap water at 60°C (140°F), rinsed with deionized water at room temperature, and dried in a circulating-air oven at 60°C (140°F). An alternate procedure for drying was also used in which the deionized water-rinsed panels were washed with absolute methanol and dried at room temperature using filtered compressed air.

Silane Primer Solution

The organosilanes were diluted to 10% pbw solids using deionized water and acidified to a pH of 3.5-5.0 with glacial acetic acid. The resulting solutions were allowed to stand 24 hours to complete hydrolysis. Silane solutions of 0.1%, 0.5%, 1.0% pbw solids were prepared by diluting aliquots with absolute methanol.

Methods of Applying Silanes

1. The diluted organosilane solutions were brushed on the metal surfaces to be bonded and dried in air.
2. The metal surfaces to be bonded were immersed in a diluted organosilane solution.
3. Neat solutions of the organosilanes were mixed in with the adhesive resin in concentrations of 0.1%, 0.5%, 1.0%.

Resins

Epon 828/Versamid 140 is a two-component epoxy adhesive system. Epon 828 is a bisphenol A epoxy resin manufactured by Shell Chemical Co., Versamid 140 is an aliphatic polyamide resin manufactured by General Mills, Inc. The latter is a combination modifier (flexibilizer) and curing agent for the adhesive system. The adhesive was formulated using 70 pbw Epon 828 and 30 pbw Versamid; it cures at 23°C (73°F) and requires approximately 0.07 MPa (10 psi) contact pressure for joint formation.

EC 2214 is a modified one-component epoxy paste manufactured by 3M Co. It is suitable at room temperature for trowel applications. It cures at 121°C (250°F) in 40 minutes. It requires only contact pressure (approximately 0.07 MPa (10 psi)) for joint formation.

Bonding Procedure

The test specimens used in this evaluation were single-lap,

shear-type specimens having 12.7 x 25.4 mm (1/2 x 1 inch) bond areas. Each was fabricated in the form of four panels and the overlap was controlled by using jigs. The surfaces to be bonded were coated with organosilane solution, air dried, the adhesive was applied and the panels assembled.

Adverse Conditions

Hot water soak/residual-strength

The bonded specimens were immersed in a thermostatically controlled deionized water bath at 60°C (140°F) for 34 days (816-hr.). The specimens were then removed in a container of water at 60°C (140°F) and transferred to the test chamber of the Baldwin Tensile Test Machine which was controlled at 60°C (140°F). Each specimen was removed from the container, placed into the grips, and tested to failure at a constant load of 16.54 MPa/min.

95% relative humidity/residual strength

Specimens were conditioned in a controlled humidity cabinet of 60°C (140°F) and 95% RH for ninety days before testing.

Conversion to SI Units

Stress units of pounds per square inch (psi) were converted to megapascals (MPa) as follows:

$$\frac{\text{psi} \times 6.8948}{1000} = \text{MPa}$$

This was in accordance with ASTM E380-74.

CONCLUSIONS

Reactive organosilane coupling agents have been demonstrated to be capable of improving the initial bond strength of epoxy-based adhesives and improving the resistance of the adhesive bonds on 2024T3 and 6061T6 aluminum alloys to moisture or water-induced degradation. The adhesive and the organosilane are most effective when the organic moieties are mutually chemically reactive and the reactivity occurs during the adhesive joint formation. The vinyl functional moiety does not react with the epoxy adhesives and is ineffective in combating joint degradation. The Z6020 (2-aminoethyl) is very reactive (fumes in air); it reacts too quickly to be effective in epoxy adhesives.

RECOMMENDATION

The specificity of reactants demonstrated, the improvements gained, and the limited scope of this investigation suggest that a long range evaluation should be conducted to determine the effects of combined elevated temperature, humidity, and stress on joints with organosilane coupling agents.

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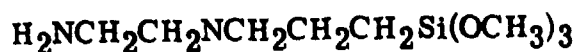
1. E. P. Plueddemann, 'Mechanism of Adhesion of Coatings Through Reactive Silanes', Journal of Paint Technology, November 1970.
2. E. P. Plueddemann, Composite Materials: Vol. 6, Interfaces in Polymer Matrix Composites, Academic Press, 1974.

Table 1

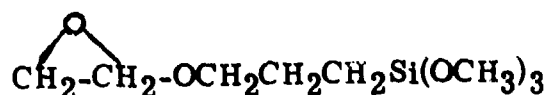
Organosilane coupling agents

Organosilanes

1. Dow Corning Z6020 - N(-2 aminoethyl)-3-aminopropyltrimethoxysilane.



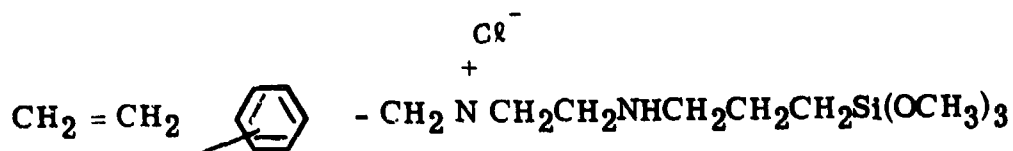
2. Dow Corning Z6040 - Glycidoxypropyltrimethoxysilane.



3. Dow Corning Z6062 - Mercaptopropyltrimethoxysilane.



4. Dow Corning Z6032 - Vinylbenzylamine functional silane.



5. Union Carbide Corp. A-1100 - N-aminopropyltriethoxysilane.



TABLE 2

SUMMARY OF ORGANOSILANE COUPLING AGENT RESULTS

ADHESIVE	SILANE PRIMER	ADHESIVE	ADVERSE EXPOSURE	SHEAR STRENGTH at 21°C (70°F)		SHEAR STRENGTH ADVERSE CONDITION		%
				M Pa	psi	M Pa	psi	
2024T3	none	EC2214	95% rh,	38.0	5510	28.7	4169	-24.3
(.063")			60°C(140°F)					
	Z6040	EC2214	90 days	41.8	6074	33.0	4783	-21.3
	Z6040	EC2214 Z6040		42.1	6107	35.7	5176	-15.0
6061T4	none	EC2214	95% rh,	21.0	3054	13.2	1914	-37.3
(.063")			60°C(140°F)					
	Z6040	EC2214	90 days	25.9	3757	25.5	3696	- 1.6
	Z6040	EC2214 Z6040		26.7	3875	28.9	4194	+ 8.
2024T3	none	Epon 828 V-140	Hot water					
(.125")			soak, 60°C	20.9	3063	22.6	3310	+ 8.0
	Z6040	Epon 828 V-140	(140°F) 34 days	22.0	3205	27.6	4000	+24.9
6061T4	none	Epon 828 V-140	"	13.2	1925	9.0	1310	-41.9
(.063")				26.1	3810	15.3	2243	-31.9
302	Z6040 (0.3%)	Epon 828 V-140	"	10.7	1565	9.6	1395	-10.8
Stainless Steel	Z6040	Epon 828 V-140		11.5	1680	10.3	1510	-10.1
	(0.3%)							

TABLE 2 (Con't)
SUMMARY OF ORGANOSILANE COUPLING AGENT RESULTS

ADHESIVE	SILANE PRIMER	ADHESIVE	ADVERSE EXPOSURE	SHEAR STRENGTH at 21°C (70°F)		SHEAR STRENGTH ADVERSE CONDITION		DEGRADATION %
				MPa	psi	MPa	psi	
2024T3	none	Epon 828 V-140	Hot water	26.9	3933	20.8	3048	-22.5
(.063")			soak, 60°C					
	Z6032 (0.3%)		(140°F) 34 days	25.9	3780	20.7	3020	-20.1
6061T4	none	Epon 828 V-140	(140°F) 34 days	11.5	1090	8.1	1180	+8.3
(.063")								
	Z6032 (0.3%)			10.5	1538	9.2	1338	-13.0
302	none	Epon 828 V-140	(140°F) 34 days	23.1	3370	19.2	2800	-3.8
Stainless Steel	Z6032			21.1	3065	20.3	2948	-0.2
2024T3	none	Epon 828 V-140	(140°F) 34 days	18.2	2648	12.6	1835	-30.7
(.125")								
	Z6020 (0.5%) (Brushed on)	Epon 828 V-140	(140°F) 34 days	20.2	2935	4.9	710	-75.9
	Z6020 (Dipped)	Epon 828 V-140	(140°F) 34 days	20.8	3025	4.2	603	-80.1
	none	Z6020		19.1	2778	12.7	1845	-33.6
	Z6020	Z6020		14.5	2180		No Bond	-100.0

TABLE 2 (Con't)
SUMMARY OF ORGANOSILANE COUPLING AGENT RESULTS

ADHEREND	SILANE PRIMER	ADHESIVE	ADVERSE EXPOSURE	SHEAR STRENGTH at 21°C (70°F)		SHEAR STRENGTH ADVERSE CONDITION		% DEGRADATION
				M Pa	psi	M Pa	psi	
2024T3	none	Epon 828 V-140	Hot water	20.5	2978	11.1	1625	-65.4
(.125")			Soak, 60°C					
	Z6062 (0.5%)		(140°F) 34 days	29.3	4255	15.6	2270	-46.7
	none	+Z6062	(140°F) 34 days	22.0	3200	14.4	2095	-34.5
	Z6062	+Z6062	(140°F) 34 days	23.6	3420	18.0	2610	-23.7
2024T3	none	Epon 828 V-140	(140°F) 34 days	19.2	2793	15.5	2245	-19.6
(.125")								
	Z6040 (0.5%)	Epon 828 V-140	(140°F) 34 days	24.8	3598	22.0	3190	-11.3
	none	Z6040		27.4	3978	13.9	2015	-49.3
	Z6040	Z6040		28.0	4080	25.5	3585	-12.1
2024T3	none	Epon 828 V-140		26.0	3778	11.0	1605	-57.5
(.125")								
	A-1100	Epon 828 V-140		18.9	2745	8.4	1221	-55.5
	(1.0%)							
2024T3	none	Epon 828 V-140		25.7	3728	11.4	1673	-55.1
(.125")								
	Z6062 (1.0%)	Epon 828 V-140		26.1	3793	12.5	1822	-52.0

APPENDIX

EFFECTS OF FUNCTIONAL COUPLING AGENTS ON BOND STRENGTHS

Effect of Epoxy Functional Coupling Agents on Bond Strengths to 2024T3 Aluminum with EC 2214

ADHEREND	SILANE PRIMER	ADHESIVE	ADVERSE EXPOSURE	SHEAR STRENGTH AT 21° (73°F)		SHEAR STRENGTH ADVERSE CONDITION		% DEGRADATION
				psi	M Pa	psi	M Pa	
2024T3	none	EC 2214	5557			4324		
(.063")			5857			4196		
			5292			4304		
			5592			4091		
			5744			3928		
			5020	5510	38.0	4169	28.7	
	Z6040	EC 2214	5783			4793		
			6134			4884		
			6155			4781		
			6241			4859		
			6421			4600		
			5709	6074	41.8	4783	33.0	
	Z6040	EC 2214	5876			5354		
		Z6040	6358			5540		
			6118			5053		
			5885			5025		
			6270			4905		
			6136	6107	42.1	5176	35.7	

Effects of Epoxy Functional Coupling Agent on Bond Strengths to 8061T4 Aluminum with EC 2214

ADHEREND	SILANE PRIMER	ADHESIVE	21°C (70°F)			SHEAR STRENGTH 95th 60°C (140°F)			AVG		
			psi	psi	M Pa	psi	psi	M Pa	psi	psi	M Pa
8061T4	none	EC 2214	2880			1959					
(.063")			3255			1869					
			3159			1796					
			2892			1963					
			3215			1984					
			2923	3054	21.0		1914	13.2			
	Z6040	EC 2214	3697			3709					
			3964			3592					
			3745			3687					
			3909			3767					
			3702			3724					
			3507	3757	25.9		3696	25.5			
	Z6040	EC 2214 26040	3788			4302					
			3909			4096					
			4052			4240					
			3729			4188					
			3866			4172					
			3908	3875	26.7		4194	28.9			

SHEAR STRENGTH

14

SHEAR STRENGTH

15

SHEAR STRENGTH

18

ADHEREND	SIANE PRIMER	ADHESIVE		SHEAR STRENGTH	
		21°C (70°F)	AVG	Hot Water Soak 60°C (140°F)	34 days AVG
1	1	1	1	1	1
2	2	2	2	2	2
3	3	3	3	3	3
4	4	4	4	4	4
5	5	5	5	5	5
6	6	6	6	6	6
7	7	7	7	7	7
8	8	8	8	8	8
9	9	9	9	9	9
10	10	10	10	10	10
11	11	11	11	11	11
12	12	12	12	12	12
13	13	13	13	13	13
14	14	14	14	14	14
15	15	15	15	15	15
16	16	16	16	16	16
17	17	17	17	17	17
18	18	18	18	18	18
19	19	19	19	19	19
20	20	20	20	20	20
21	21	21	21	21	21
22	22	22	22	22	22
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71	71	71	71	71	71
72	72	72	72	72	72
73	73	73	73	73	73

17

[illegible]

SHEAR STRENGTH

18

**Effects of Diamino Functional Coupling Agent on Bond
Strengths to 2024T3 Aluminum using Epon 828/Versamid 140**

ADHEREND	SILANE PRIMER	ADHESIVE	21°C (70°F) AVG				SHEAR STRENGTH Hot Water Soak 60°C (140°F) 34 days			
			psi	psi	M Pa	psi	psi	psi	M Pa	AVG
			psi	psi	M Pa	psi	psi	psi	M Pa	AVG
2024T3	none	Epon 828 V-140	2700			1800				
			2500			2000				
			2630			1640				
			2760		18.2	1900		1835	12.6	
	.5%									
	Z6020	Epon 828 V-140	2930			730				
	Brushed		2900			660				
	on		2950			730				
			2960	2935	20.2	720	710		4.9	
	Z6020		2960			600				
	Dipped into		2900			500				
	Silane		3150			650				
	Solution		3090	3025	20.8	660	603		4.2	
	none	1% Z6020	2790			1730				
			2810			1860				
			2760			1940				
			2750	2778	19.1	1850	1845		12.7	

[illegible]

Effects of Mercapto Functional Coupling Agent on Bond Strengths to 2024T3 Aluminum using Epon 828/Versamid 140

ADHEREND	SLAKE PRIMER	ADHESIVE	SHEAR STRENGTH				Hot Water Soak			
			21°C (70°F)				60°C (140°F) 34 days			
			psi	psi	M Pa	AVG	psi	psi	M Pa	AVG
2024T3	none	Epon 828 V-140	2810				1630			
			2900				1630			
			3100				1560			
			3100	2978	20.5		1680	1625	11.1	
	.5%									
	26062	Epon 828 V-140	4250				2220			
	Brushed		4270				2300			
	on		4140				2320			
			4360	4255	29.3		2240	2270	15.6	
	none	1% Z6062	3100				2060			
			3100				1980			
			3300				2160			
			3300	3200	22.0		2180	2095	14.4	
	26062	1% Z6062	3400				2620			
			3450				2580			
			3410				2580			
			3420	3420	23.6		2660	2610	18.0	

Effects of Epoxy Functional Coupling Agent on Bond Strengths to 2024T3 using Epon 828/Versamid 140

ADHEREND	SILANE PRIMER	ADHESIVE	21°C (70°F) AVG				SHEAR STRENGTH Hot Water Soak 60°C (140°F) 34 days AVG			
			psi	psi	M Pa	psi	psi	M Pa		
2024T3	none	Epon 828 V-140	2860			2120				
(.125")			2730			2260				
			2780			2280				
			2800	2793	19.2	2320	2245	15.5		
	.5%									
	Z6040	Epon 828 V-140	3520			3120				
	Brushed on		3320			3100				
			4000			3140				
			3550	3598	24.8	3400	3190	22.0		
	none	1% Z6040	3860			2000				
			4130			2100				
			3990			1960				
			3930	3978	27.4	2000	2015	13.9		
	Z6040	1% Z6040	4060			3540				
			4070			3540				
			4050			3580				
			4140	4080	28.0	3560	3585	25.5		

[illegible]

SHEAR STRENGTH
Hot Water Soak
60°C (140°F) 34 days AVG

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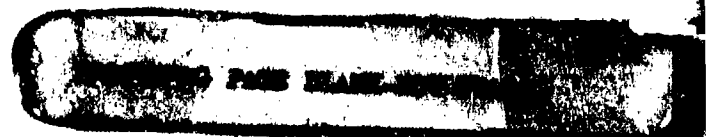
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